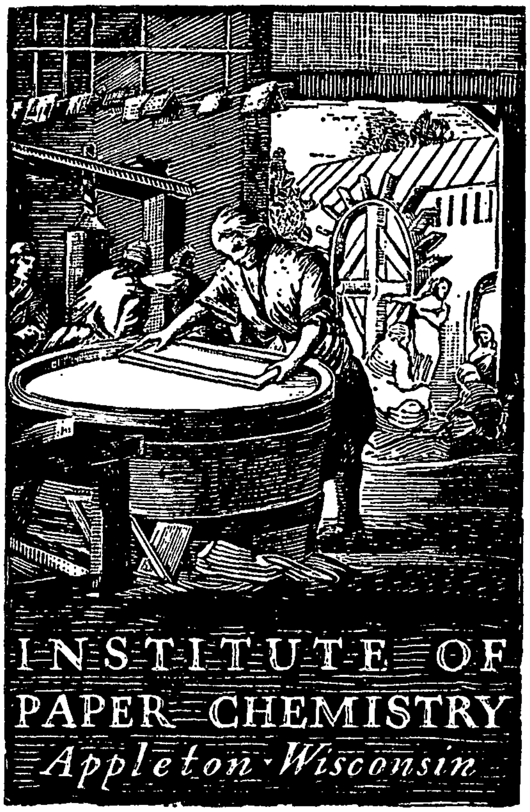

FILTRATION RESISTANCE AND ITS COMPONENTS

THE EFFECT OF CELL WALL THICKNESS

Project 2406

Report Eight

A Progress Report

to

MEMBERS OF GROUP PROJECT 2406

March 13, 1967

THE INSTITUTE OF PAPER CHEMISTRY
Appleton, Wisconsin

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TABLE OF CONTENTS

	Page
SUMMARY	1
INTRODUCTION	2
METHOD OF APPROACH	5
EXPERIMENTAL PROGRAM	10
FILTRATION DATA SUMMARY	17
RESULTS AND DISCUSSION	21
ACKNOWLEDGMENT	29
LITERATURE CITED	30
APPENDIX I. CARLETRAN PROGRAM FOR VARIABLE SPECIFIC VOLUME	31
APPENDIX II. AVERAGE SPECIFIC SURFACE AND SPECIFIC VOLUME	33

TABLE OF CONTENTS

	Page
SUMMARY	1
INTRODUCTION	2
METHOD OF APPROACH	5
EXPERIMENTAL PROGRAM	10
FILTRATION DATA SUMMARY	17
RESULTS AND DISCUSSION	21
ACKNOWLEDGMENT	29
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THE INSTITUTE OF PAPER CHEMISTRY
Appleton, Wisconsin

FILTRATION RESISTANCE AND ITS COMPONENTS

THE EFFECT OF CELL WALL THICKNESS

SUMMARY

The curvature observed in the rectification of constant-rate filtration data to yield specific surface and specific volume has led to the hypothesis of a pressure-dependent specific volume. Based on this hypothesis, it was speculated that the pressure dependency of the specific volume could be related to fiber conformability.

The experimental program designed to evaluate this test for an index of conformability has not yielded the anticipated results. The test is not sensitive enough to compare the response of fiber types, and the theory has not been developed rigorously to the point where analytical comparisons may be made.

INTRODUCTION

The conformability or deformability of wood pulp fibers has not been measured with any degree of reliability with existing equipment or techniques. In an attempt to develop a measure of this property, it was felt that the concept of a specific volume which was pressure dependent might have some utility in that the structural elements comprising the fiber cell wall should affect the rate of response of the swollen volume to a deforming pressure.

Conformability, deformability, or flexibility are terms applied to describe that property of cellulosic fibers whereby the fibers are brought into intimate contact to establish bonding or a high degree of mechanical entanglement in wet fiber webs. This work was designed to evaluate a particular phenomenon and the choice of nomenclature is arbitrary.

The complex morphology of the naturally occurring cellulosic fiber as used in the pulp and paper industry makes it difficult if not impossible to predict the water-swollen specific volume from any dimensional measurements. These fibers are comprised of several structural elements in various layers, and each layer may contain material which is chemically unique in its composition and response to water. The existence of pits and pores plus a lumen through the center of the fiber further complicates the mensuration of a swollen fiber.

During the past decade, a technique has evolved through the work of Ingmanson (1-3) which has given reliable values of the swollen specific volume defined in the hydrodynamic sense as the volume denied to flow per unit mass of dry cellulose during a permeation or filtration process. This definition encompasses the volume of the cellulose plus the volume of any immobilized water associated with the fiber. For present considerations it is not important how or where the water is immobilized.

If Ingmanson's constant-rate filtration technique is used to evaluate the swollen specific volume, as the pressure-drop increases the compacting forces could express water from the fibers and a changing volume would result. The stresses in the mat are the result of fluid drag forces which act over the surface of the fiber, impeding flow. The fibers are supported at a point within the mat structure, and as a result there would be a stress intensification at the points of contact.

In such a filtration process the drag forces are cumulative, each successive layered element contributing to the total and supporting all loading from the upstream elements. Thus, the maximum stress will occur in those fibers which are in the bottom of the mat at the septum interface. The conditions of the constant-rate test have been established such that a maximum overall pressure drop of 10^5 dynes/cm.^{2} is sustained at the septum.

Swanson and coworkers (4) have established that the porosity of the cell wall could be as high as 0.5. Thus, if the pores are sufficiently large, water could be expressed from the walls and from the lumen through the walls. Therefore, the application of fluid or mechanical compacting pressure could result in a changing specific volume whose sensitivity to change may be dependent upon the structural form of the fiber cell wall.

Major differences in morphology may be observed in the springwood *vs.* summerwood fibers of a given species. The springwood will have a thin cell wall and large lumen, and the summerwood a thick cell wall and small lumen. The differences are quite apparent in Fig. 1, which is a photomicrograph of a cross section of Douglas-fir.

If samples of pulp were prepared from each of these two fiber types, it might be possible to attribute rate responses of specific volume to structural

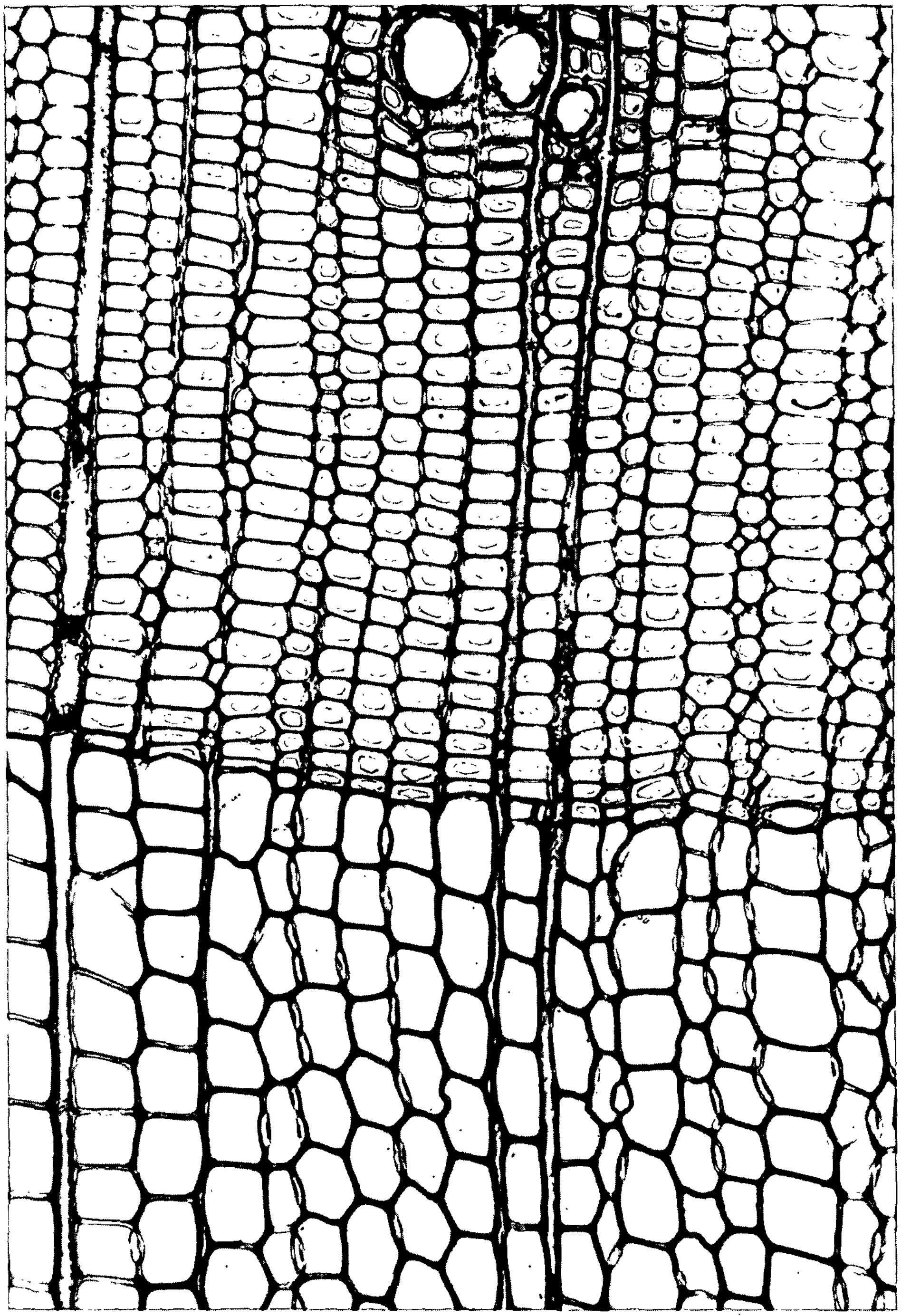

Figure 1. Photomicrograph of Cross Section of Douglas-Fir

forms of the fibers. Using laboratory semimicropulping procedures and hand chipping tools, sufficient sample for hydrodynamic characterization can be prepared. The same wood source was used in other phases of the project, notably the single fiber work.

METHOD OF APPROACH

Initially, this work was inaugurated with the primary objective of evaluating the filtration resistance component of a pressure-gradient-dependent specific volume as a pulp evaluation tool and to demonstrate current calculational techniques.

Ingmanson's development of a constant-rate filtration procedure to evaluate the specific resistance of pulp fibers has been a significant contribution in terms of drainage and fiber properties (3). The resistance is defined by Darcy's law in the form applicable to compressible media as

$$R = \frac{A^2}{\mu C q^2} \quad \frac{\Delta P}{\theta} = B \frac{\Delta P}{\theta} \tag{1}$$

where

R = specific filtration resistance, cm./g.,

A = cross-sectional area of flow, cm.^{2},

μ = fluid viscosity, poises,

C = mass of fiber deposited per unit volume of filtrate, g./cc.,

q = volumetric flow rate, cc./sec.,

θ = filtration time, sec.,

ΔP = frictional pressure drop across fiber mat, dynes/cm.^{2}, and

B = filtration constant, $A^2/\mu C q^2$, c.g.s. units.

Filtration resistance may be separated into its components of specific surface, specific volume, and compressibility where the compressive behavior is determined in a separate test. The current form of the function used to evaluate specific surface and specific volume has evolved from academic and sponsored research. The equation is a modification of the well-known Kozeny-Carman equation (5) in its integrated form (6):

$$\frac{\Delta P}{\theta C_p^{1/2}} = \frac{3.5(1 - N/2)S_w^2}{Bv^{1/2}} [1 + 57v^3 (1 - N/2)^6 C_p^3] \qquad (2)$$

where

$\underline{S_w}$ = fiber specific surface, cm.^{2}/g.,

$\underline{v}$ = fiber specific volume, cc./g.,

$\underline{C_p}$ = bed density, g./cc., and

$\underline{N}$ = empirical constant, dimensionless.

The compressibility is determined by a separate static experiment with the data being correlated in the form of the exponential relationship:

$$C_p = MP^N \qquad (3)$$

where $\underline{M}$ and $\underline{N}$ are empirical constants and $\underline{P}$ is a uniform mechanical compacting load in dynes/cm.^{2}.

The data of a filtration experiment on a softwood sulfite pulp have been fitted to Equation (2) and are shown in Fig. 2. The pronounced curvature of the experimental data points is obvious and the dashed line represents the best fit over the range of from 30 to 90 cm. of water.

Meyer in 1962 (7) attributed the general curvature observed in the filtration data of pulp slurries to a variable specific volume. It was Meyer's

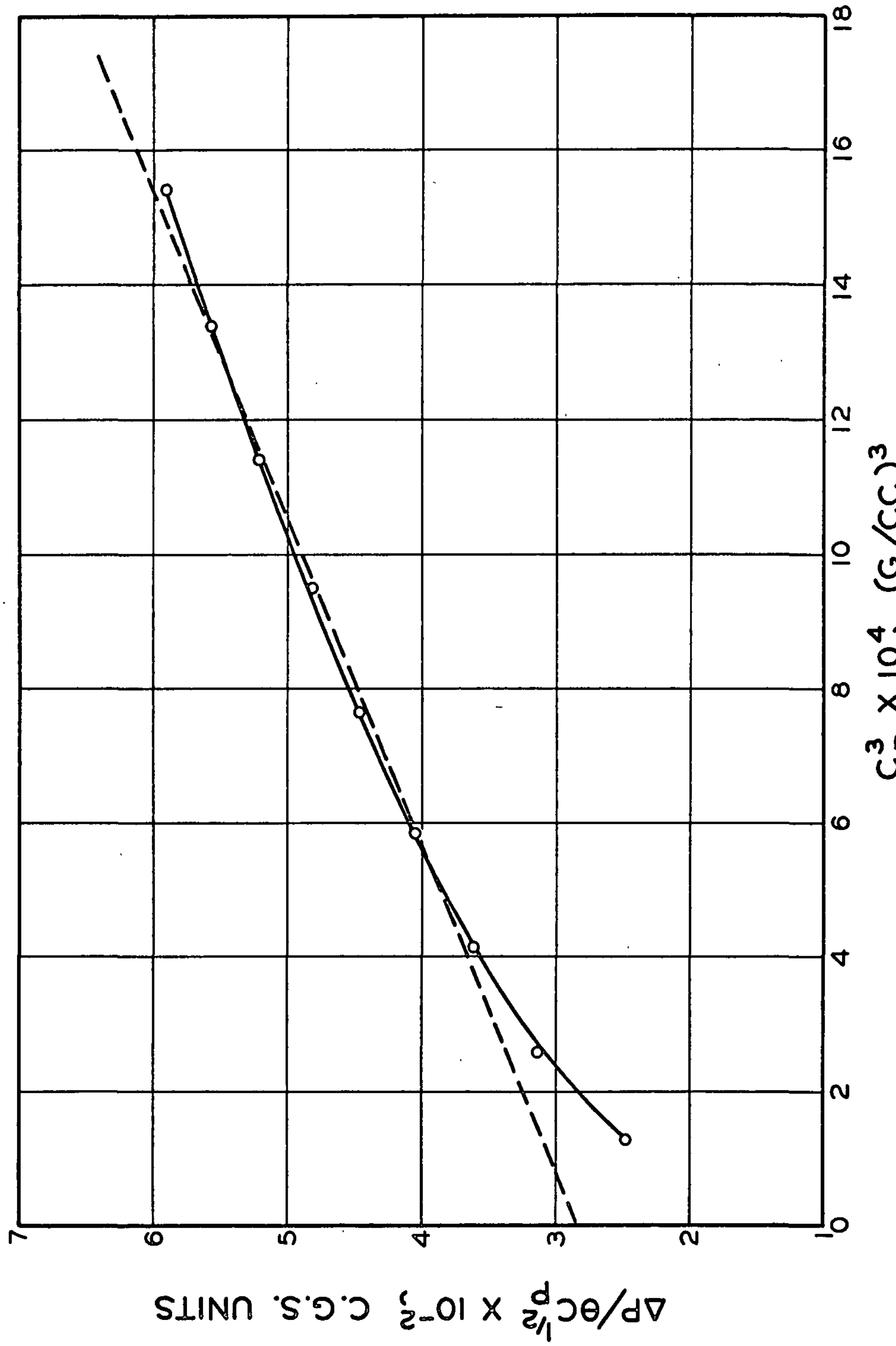

Figure 2. Rectified Constant-Rate Filtration Data

contention that the stresses encountered in a fiber mat being formed during a filtration process could be great enough to cause deformation with the resultant expression of water from the fiber.

Although it was recognized that Meyer's hypothesis had physical significance, the necessary mathematical procedures had not been utilized to evaluate a variable $\bar{v}$. Recently, he has been able to apply the Lagrangian interpolation process to Equation (2) to yield an approximate solution for the case of a variable specific volume (8).

Rewriting Equation (2) in the simple algebraic form of a linear relationship,

$$y = mx + b \tag{4}$$

where y is $\Delta P/\theta C^{1/2}$, x is $C_p^{\;3}$, and b is the intercept and m the slope, it is possible to simplify the form of Equation (2) such that the appropriate identities may be substituted into the Lagrangian equation. Given

$$y = y_1 \frac{(x - x_2)\,(x - x_3)\ldots(x - x_n)}{(x_1 - x_2)\,(x_1 - x_3)\ldots(x_1 - x_n)} + y_2 \frac{(x - x_1)\,(x - x_3)\ldots(x - x_n)}{(x_2 - x_1)\,(x_2 - x_3)\ldots(x_2 - x_n)} + \ldots + y_n \frac{(x - x_1)\,(x - x_2)\ldots(x - x_{n-1})}{(x_n - x_1)\,(x_n - x_2)\ldots(x_n - x_{n-1})} \tag{5}$$

we may now solve for any point along the curve shown in Fig. 2. Although the computational procedure is lengthy, a solution may be obtained for a limited number of points by manual calculations. The number of points is, of course, established by the size of the increment chosen in Δx. Thus, by setting an arbitrary size to Δx, the increment over which the average value of $\bar{v}$ is to be calculated is set. For any two successive points (y, y_1) and (x, $x + \Delta x$), the equation of a straight line is satisfied and the identities of Equation (2) may be substituted to yield the values of the specific surface and specific volume. The pressure drop at which $\bar{v}$ is to be reported is calculated as the average value for the x and $x + \Delta x$ increment from the compressibility equation.

Equation (5) may be solved for virtually an infinite number of points on a computer. Using the machine language known as Carletran, such a computer program has been prepared and is illustrated in Appendix I.

White (9), in an experimental program to evaluate specific volume of cellulosic fibers independent of hydrodynamic or mensuration techniques, concluded that springwood-summerwood fibers had markedly different specific volumes. He also observed that the cell wall structure appeared to influence the movement of liquid water in the fiber lumen.

Substantiated by White's observations, it was concluded that springwood-summerwood fibers of a softwood species would give major morphological differences and that the springwood fiber should have a significantly larger swollen specific volume. These two fiber systems would therefore give samples which should have different responses to pressure gradients because of the fiber structural components and markedly different levels of swelling.

Previous correlations (10) of specific volume *vs*. pressure drop had in general fit the form of an exponential relationship

$$\bar{v} = a\Delta P^{-b} \qquad (6)$$

where ΔP is the frictional pressure drop and a, b are empirical constants. It must be stressed that Equation (6) is empirical in nature.

Preliminary analysis indicated the possibility that the absolute numerical value of the constant b might be related to the fiber's conformability.

Therefore, it was anticipated that as b becomes larger the fiber would be more deformable, and on this basis it would be predicted that a springwood fiber should exhibit a larger b value or greater conformability.

EXPERIMENTAL PROGRAM

A section of a Douglas-fir log was selected to prepare the necessary fiber samples for this work. The separation of the springwood-summerwood fibers is quite distinct and each type is relatively plentiful in the annual growth rings.

The log was cut into approximately 1-inch thick rings which were then broken into semirectangular sections. These sections were placed under the knife of a hand chipper and very carefully cut to yield only a single type of fiber in each of the successive sections. Sufficient material was prepared in this fashion to provide an adequate supply of pulp sample for characterization.

The springwood and summerwood chip samples were each divided evenly into two batches for separate pulping schemes. One set, comprising a sample of each type of fiber, was to be treated by a mild chloriting process and the other set by a conventional kraft cook.

The two samples to be treated by the chloriting were placed in vacuum desiccators filled with the chloriting solution. The chemical concentration was based on 100% active chemical on the wood weight. The samples were treated at room temperature for approximately 2 weeks and were then caustic extracted with a solution of 2.5% NaOH, acid washed, caustic treated, and acid washed again. The yields of this sample were 46 and 57.3% for the springwood and summerwood, respectively.

The other sample was pulped by a kraft cook in small laboratory digesters. This pulp was then given the standard chlorine dioxide bleaching sequence.

The yields of this set of samples were 35.9 and 41.2% for the springwood and the summerwood, respectively.

As part of the planned experimental program, the fibers were characterized as completely as possible to obtain pertinent dimensions. These examinations included length distributions, cell wall thickness, cross sections (wet and dry mount), fiber width, lumen width, and weight factor determinations (as used in fiber analysis).

Table I gives the results of the fiber microscopy examinations and Fig. 3-6 are photomicrographs of representative cross sections (wet mount) for each fiber and chemical treatment.

TABLE I

FIBER PROPERTIES

	Springwood		Summerwood	
	Chlorited	Kraft	Chlorited	Kraft
Weight factor	1.23	0.97	1.90	1.42
Weighted average length, mm.	2.00	2.06	2.55	2.46
Arith. average length, mm.	1.33	1.48	1.91	1.74
Fiber width, microns	64.23	61.06	37.30	32.60
Cell wall thickness, microns	2.49	2.30	6.74	5.21
Lumen width, microns	59.25	56.46	23.82	22.18

Attempts were made to evaluate the relative contribution to the cross-sectional area of each portion of the fiber. Making tracings of the cross sections and using a planimeter, the differences between total area and lumen area were evaluated. However, the embedding technique apparently collapses at least a portion of the lumen, and the microtoming procedure apparently causes some degree

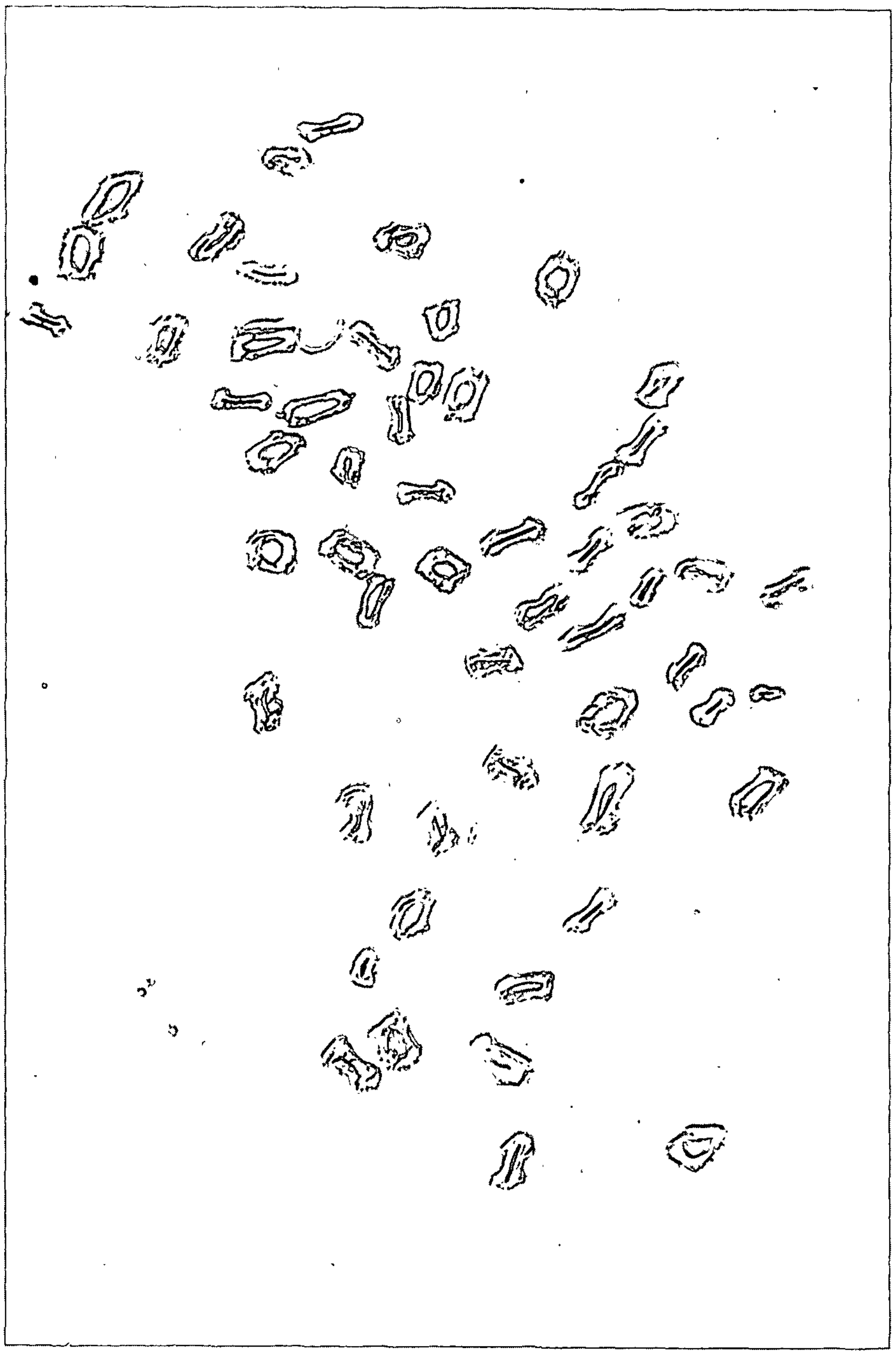

Figure 3. Photomicrograph of Cross Section of Kraft Summerwood Fiber

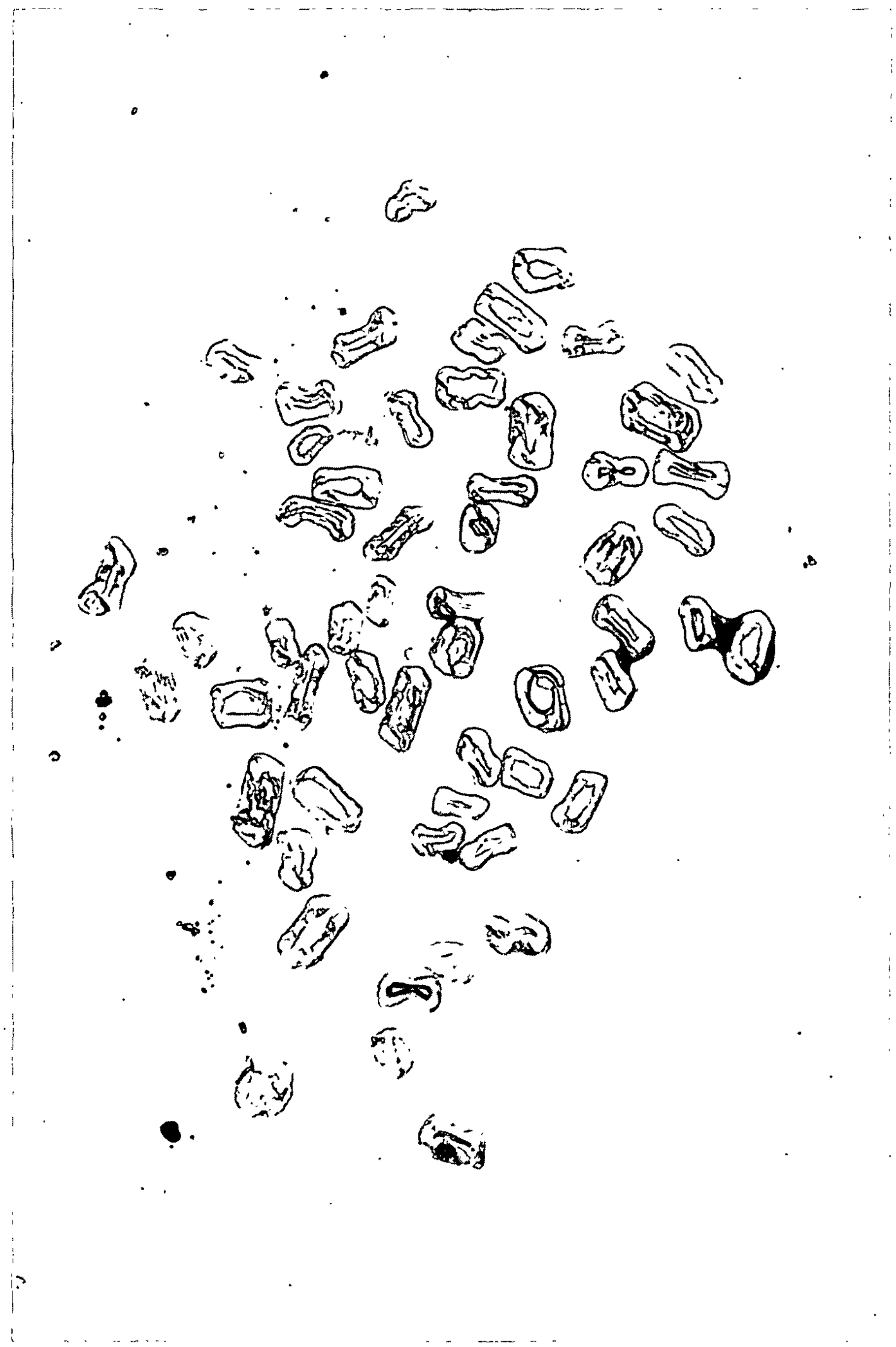

Figure 4. Photomicrograph of Cross Section of Chlorited Summerwood Fiber

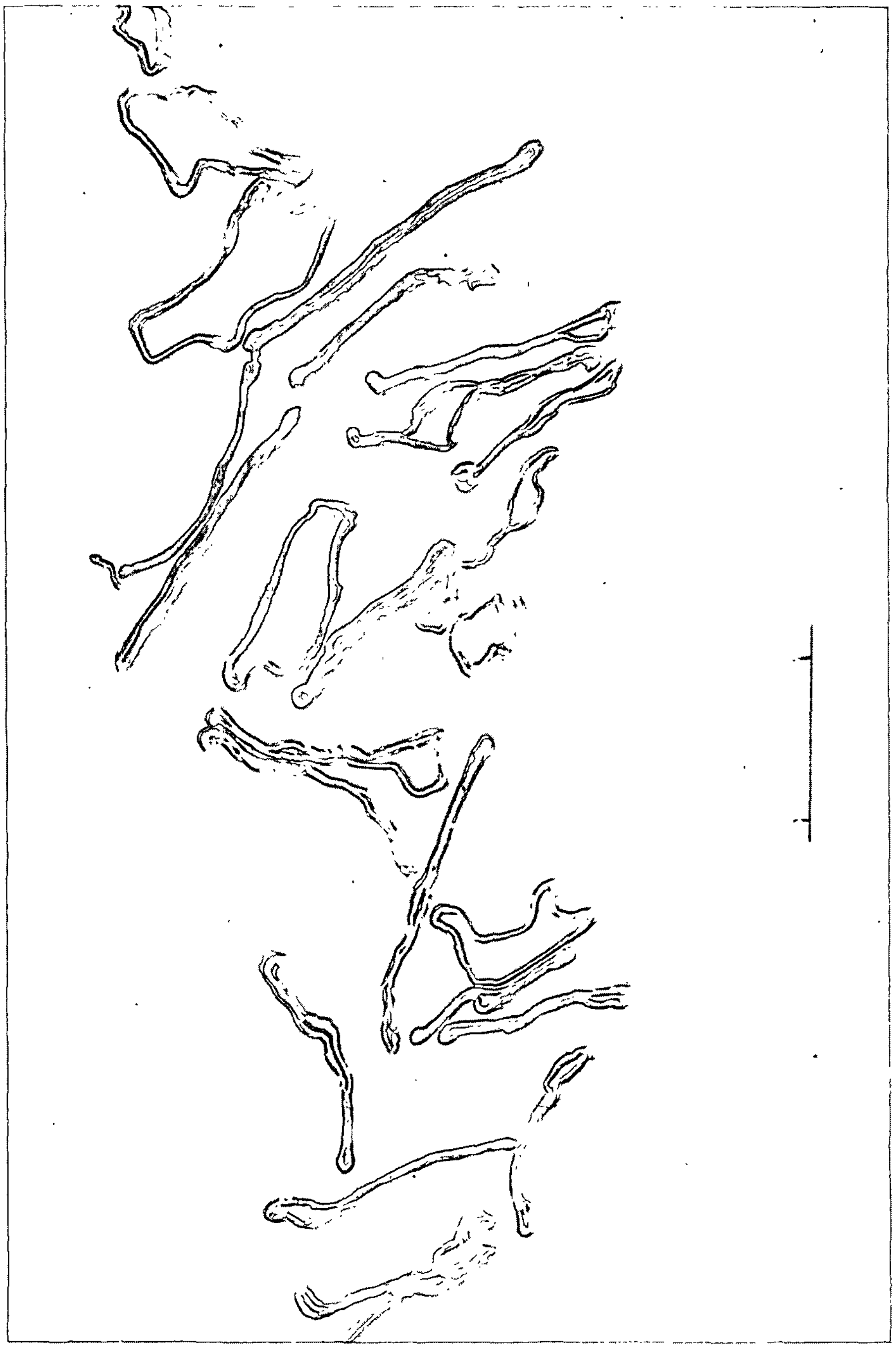

Figure 5. Photomicrograph of Cross Section of Kraft Springwood Fiber

Figure 6. Photomicrograph of Cross Section of Chlorited Springwood Fiber

of distortion. These factors greatly influenced the results, and it became apparent that calculations based on these measurements might not be even the right order of magnitude.

The photomicrographs shown in Fig. 3-6 do serve to illustrate the marked differences between the springwood and summerwood. The summerwood fibers tend to be oval in cross section and indicate a rather surprising uniformity in dimensions. The springwood fibers are to a large extent completely collapsed and resemble ribbons or elliptical fibers with a very high aspect ratio.

At the present time, techniques have not been refined to the point where water-saturated fibers can be embedded for cross sectioning. Unfortunately, any drying or solvent exchange tends to collapse or change the dimensions which are critical for predicting a changing specific volume based on a percentage decrease in lumen area.

An effort was made to obtain a definition of the degree of collapse by wet and dry mounting in an epoxy and/or in methacrylate, which exhibits a high degree of shrinkage upon curing. The precision of the measurements was so unsatisfactory, however, that no definitive conclusions could be reached.

FILTRATION DATA SUMMARY

A series of carefully controlled constant-rate filtrations were performed on the pulp samples (3). Table II lists the filtration conditions for all the samples.

TABLE II

FILTRATION CONDITIONS

Sample	Flow Rate, cc./sec.	Consistency, g./cc. x 10^4	Temp., °C.	Filtration Constant, $B \times 10^{-8}$
Summerwood (chlorited)	81.8	2.08	23.7	1.59
Springwood (chlorited)	54.3	1.00	25.9	7.99
Summerwood (kraft)	81.8	1.08	26.7	3.23
Springwood (kraft)	40.5	1.01	25.9	14.1

The filtration resistances are given in Table III and are shown in the standard representation in Fig. 7 and 8. The curvature in the specific resistance *vs*. pressure plot is due to the compressible behavior of the fibers. The level of the resistance reflects the contribution of the surface area as the resistance is proportional to the square of the specific surface.

The compressibility functions established for the four pulp samples are given in Table IV. There is a marked difference in the response not only of each fiber type but also of each cooking sequence. The constants were established from the calculated line of best fit of the compressibility measurements.

TABLE III

FILTRATION RESISTANCE

Pressure Drop, $\Delta P/\rho g.$, cm. water	Chlorited		Kraft	
	Sp. Wood, $R \times 10^{-8}$ cm./g.	Summerwood, $R \times 10^{-8}$ cm./g.	Sp. Wood, $R \times 10^{-8}$ cm./g.	Summerwood, $R \times 10^{-8}$ cm./g.
10	0.60	0.092	0.78	0.132
20	0.87	0.116	1.15	0.168
30	1.13	0.134	1.44	0.194
40	1.36	0.147	1.71	0.214
50	1.58	0.158	1.96	0.232
60	1.80	0.169	2.20	0.251
70	2.02	0.180	2.45	0.268
80	2.26	0.189	2.68	0.285
90	2.48	0.197	2.91	--

TABLE IV

COMPRESSIBILITY CONSTANTS[a]

	M		N	
	Chlorited	Kraft	Chlorited	Kraft
Springwood	0.000785	0.00127	0.437	0.396
Summerwood	0.0015	0.00193	0.383	0.362

[a] Compressibility equation $c = MP^N$ where M and N are empirical constants and P is the compacting pressure in dynes/cm.^{2}

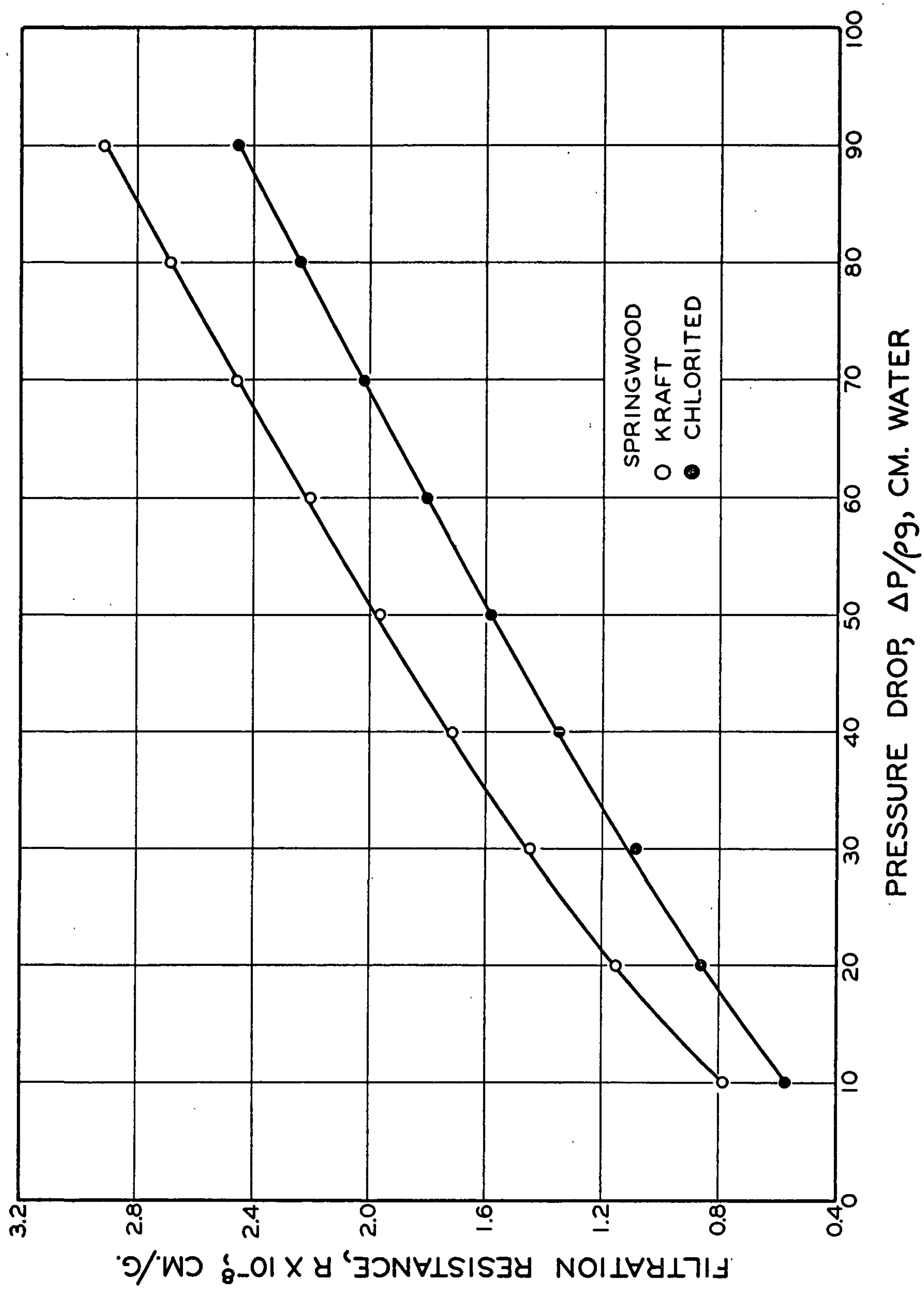

Figure 7. Filtration Resistance as a Function of Pressure Drop

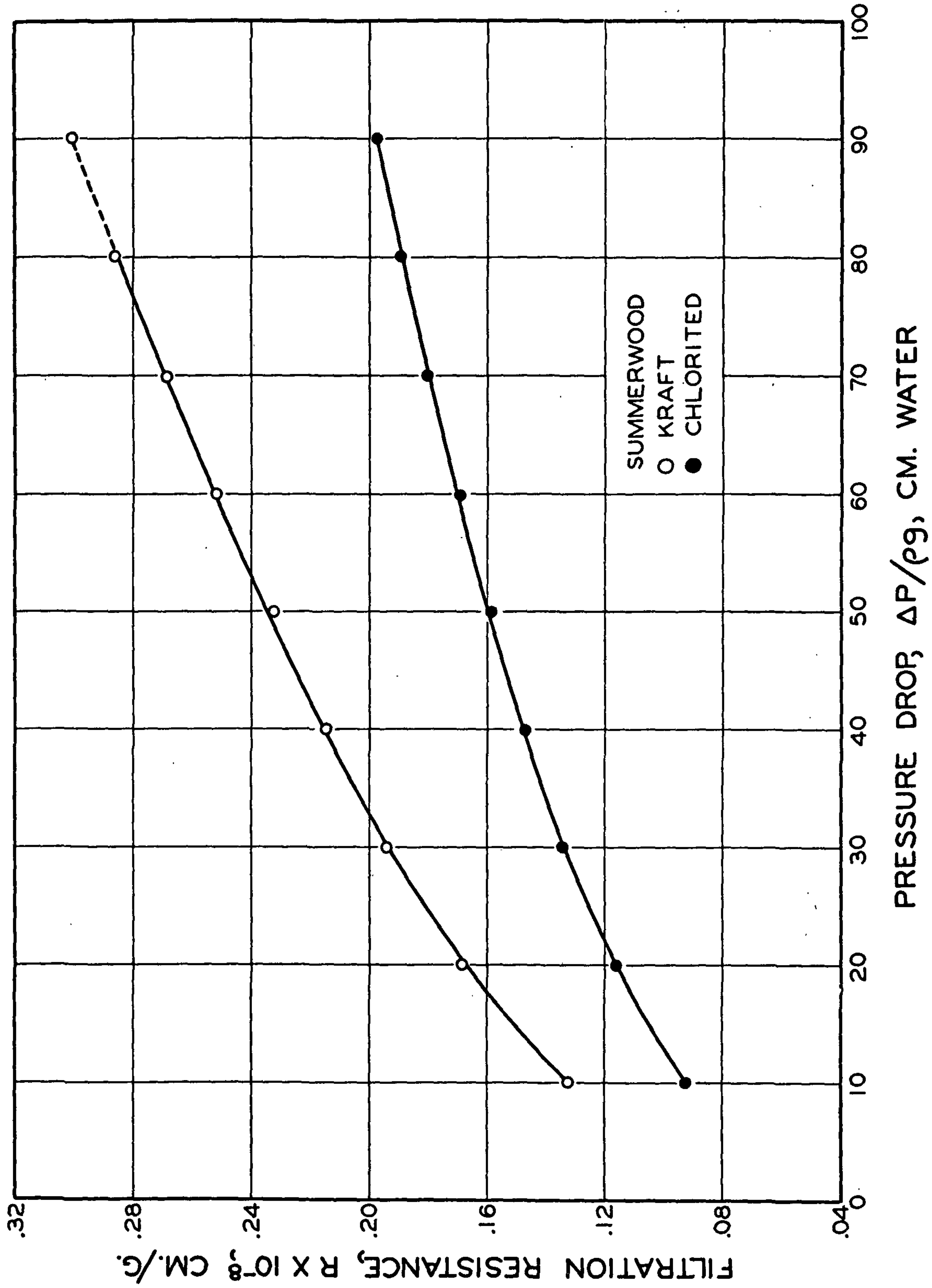

Figure 8. Filtration Resistance as a Function of Pressure Drop

RESULTS AND DISCUSSION

Using the constant-rate filtration data and the computational technique developed by Meyer (8), correlations were developed, as shown in Fig. 9 and 10, which give an arbitrary representation of a changing specific volume as a function of pressure drop. The exponential function appears to be the best representation of the data to date, although this may be modified as additional refinements in technique are developed. Appendix II gives the tabulated specific surface and specific volumes calculated by the original form of Equation (2).

The unusual sinusoidal character of the plotted points apparently stems from minor errors in the raw data. The calculational procedure has been carefully checked by computing the expansion of the series representing e^x and agrees with the tabulated data to within the significant figure of the entry. Also, the sine wave function was tested, and it also agrees within the tabulated values. Therefore, it has been argued that the snaking characteristic of the curve must stem from small errors in the data which are beyond the normal precision expected of such a test.

Since the scatter of the points appeared to be such that no simple eye fitting of a straight line could be justified, the equation of the line of best fit was calculated by an algebraic averaging technique. The form of the function was assumed to be

$$v = aP^{-b} \qquad (15).$$

The original premise in designing this program for comprehensive fiber characterization and constant-rate filtration experiments had been that fibers with markedly different cell wall structure should exhibit markedly different rates of response of specific volume to compacting pressure, thus indirectly

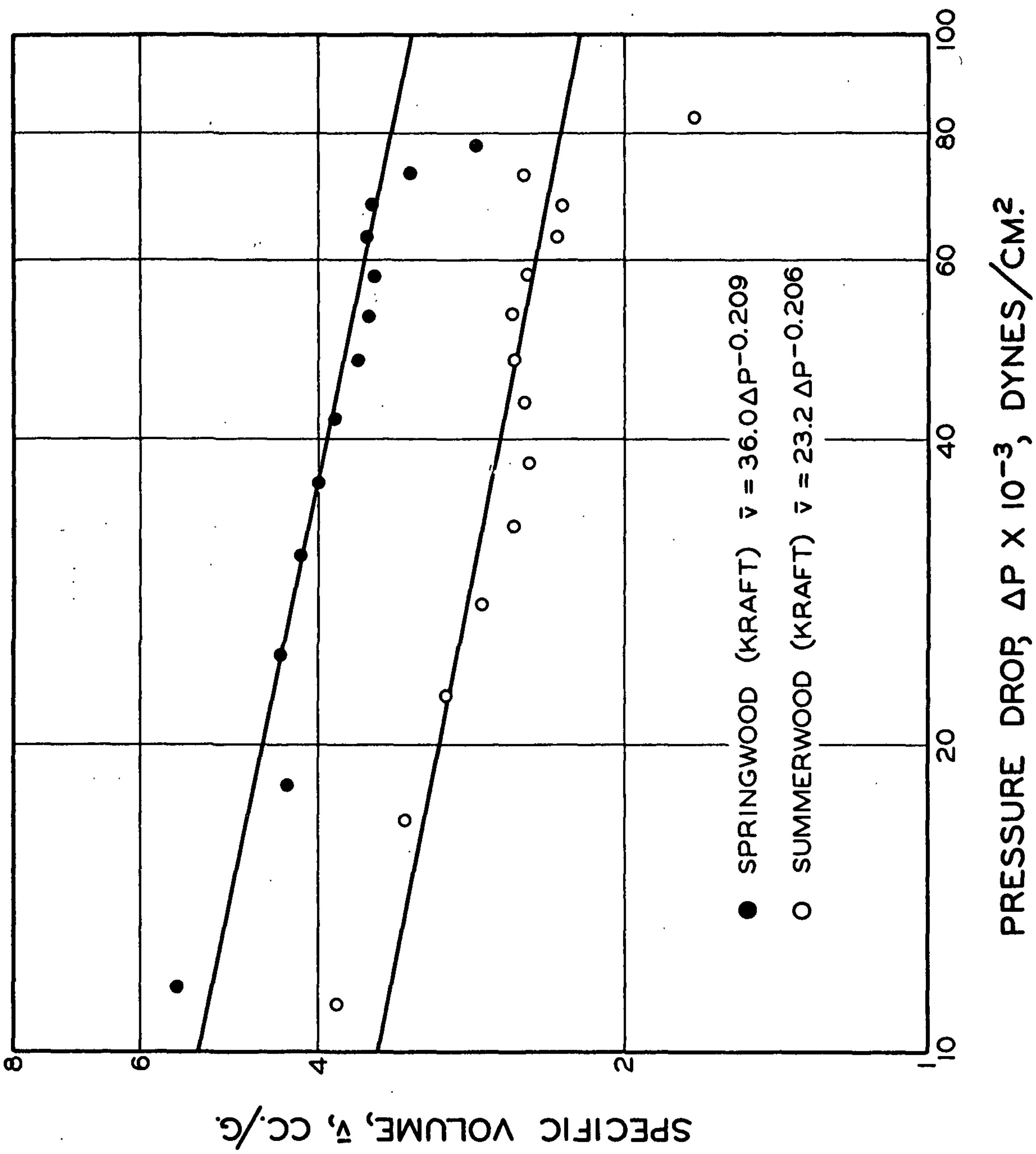

Figure 9. Specific Volume $\bar{v}$ as a Function of Compacting Pressure

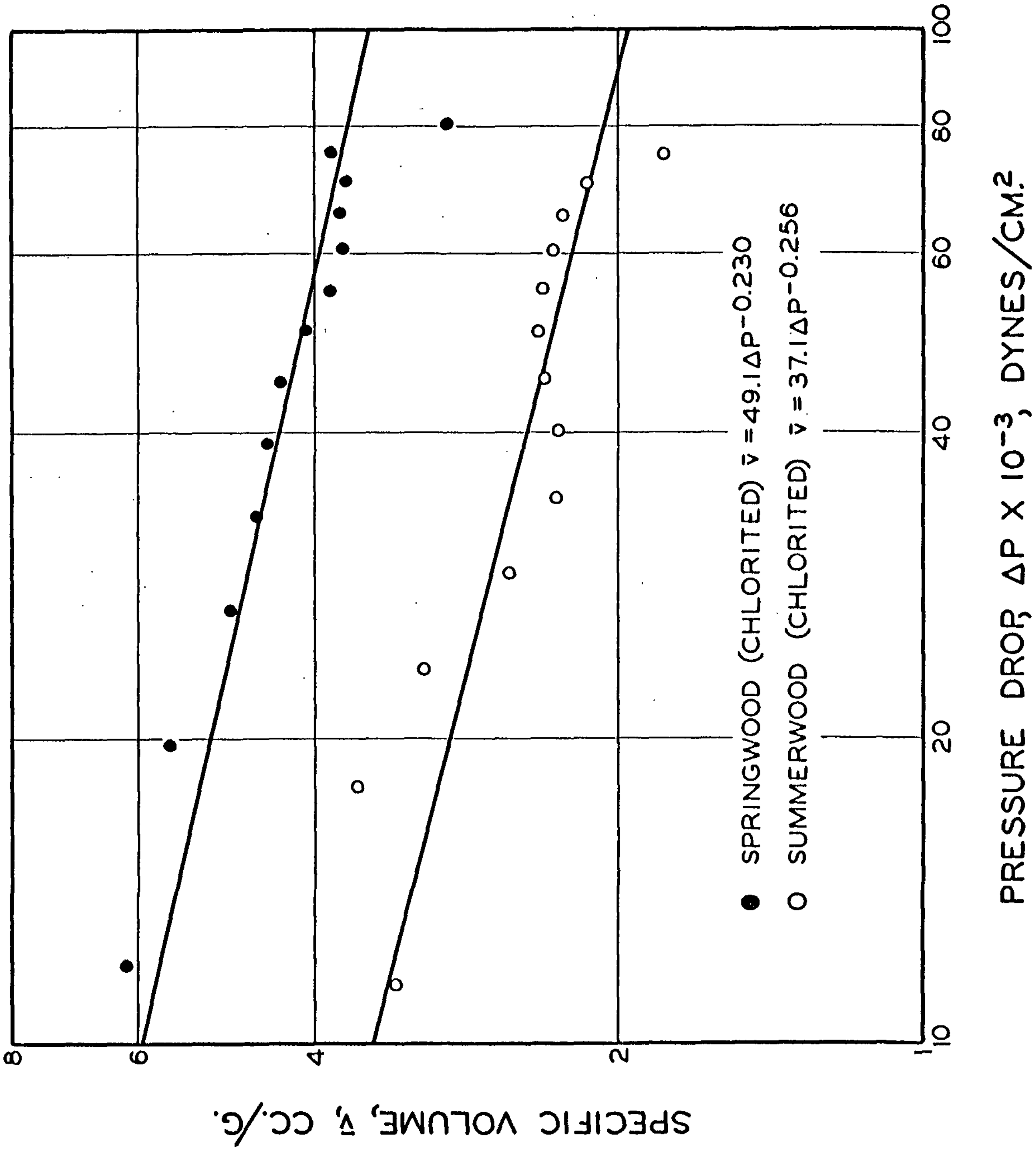

Figure 10. Specific Volume $\underline{\overline{v}}$ as a Function of Compacting Pressure

giving an index of fiber deformability. It was anticipated that the fiber morphology and mechanical or chemical treatment could thus be characterized as to their effects on the fiber conformability. It was anticipated that the curves shown in Fig. 9 and 10 would tend to converge thus substantiating the original premise in designing the experiments.

Although the original argument of the rate of response of specific volume to pressure being affected by fiber structure may still be valid, there are too many possible mechanisms of fiber collapse to permit analysis of the data in any simple, straightforward fashion. The inherent deficiencies in the theory for application to this case further complicates the situation.

There are, at least at present, three possible forms the specific volume versus pressure drop function for springwood-summerwood fibers may take. These are broadly defined as:

1. Convergence. The slope of the springwood is greater than that of the summerwood, and over the range of the filtration pressure data the curves converge.

2. Divergence. The slope of the summerwood curve is slightly greater than that of the springwood.

3. Parallelism. The curves are parallel over the pressure drop range.

Each type of relationship is illustrated in Fig. 11-13 for hypothetical fiber systems of springwood-summerwood.

The curves of Fig. 11-13 were prepared to represent a springwood-summerwood fiber system. The plots were originally prepared on a log-log representation and then transposed to the liner coordinate system. The linear plots will illustrate that eventually all three possibilities must converge to the psychometrically determined volume.

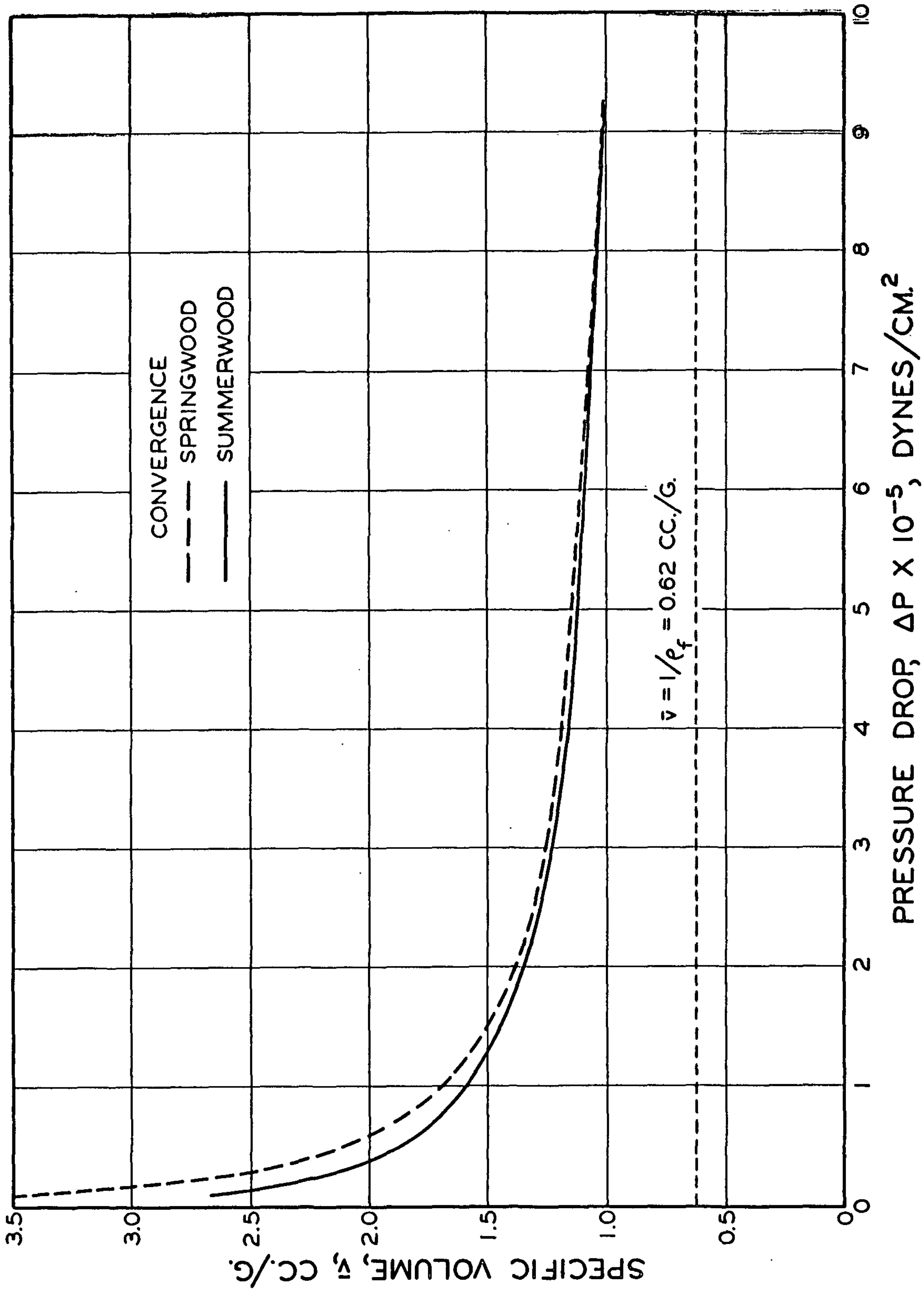

Figure 11. Convergence of Specific Volumes for a Hypothetical Set of Fiber Samples

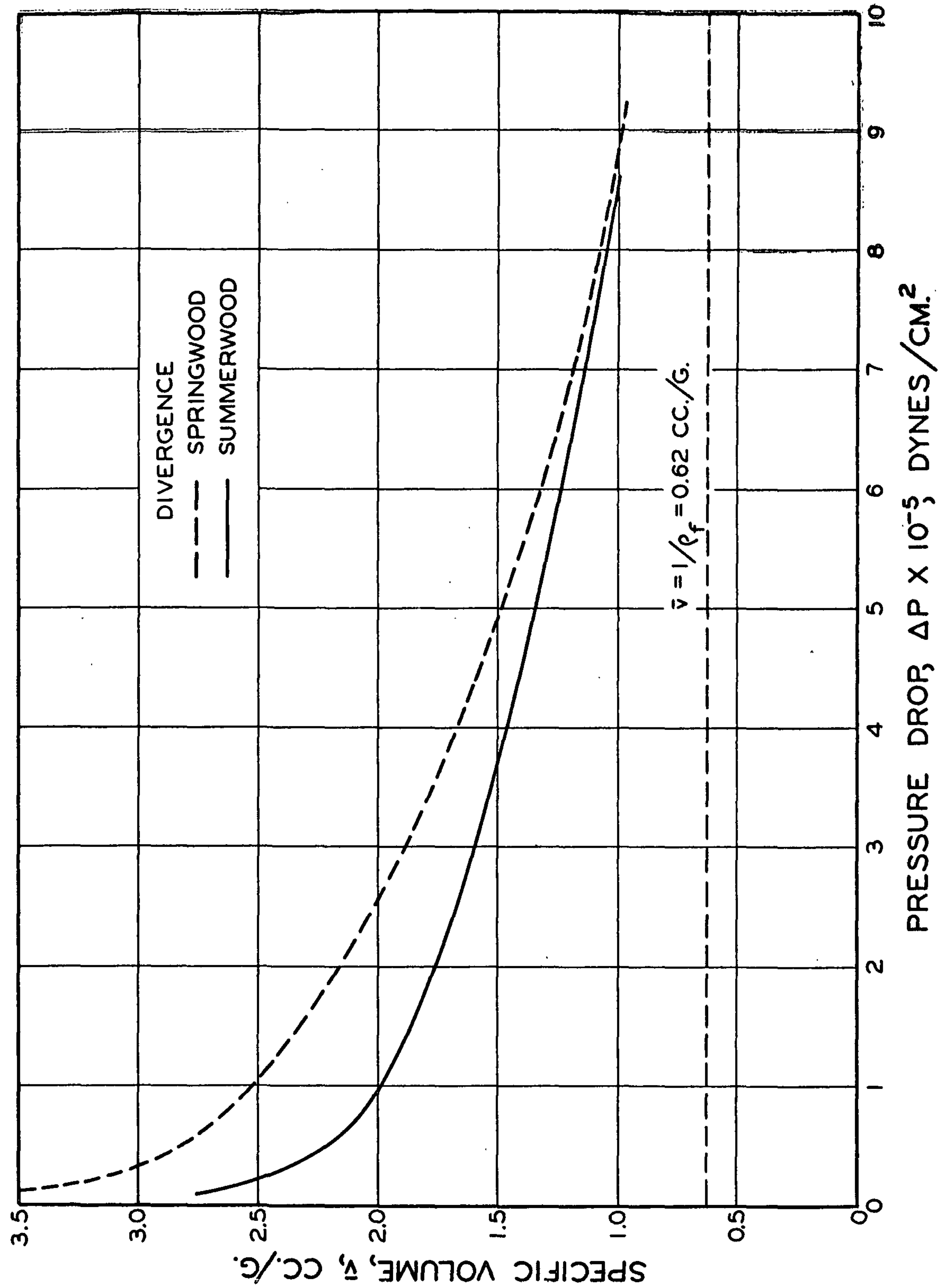

Figure 12. Divergence of Specific Volumes for a Hypothetical Set of Fiber Samples

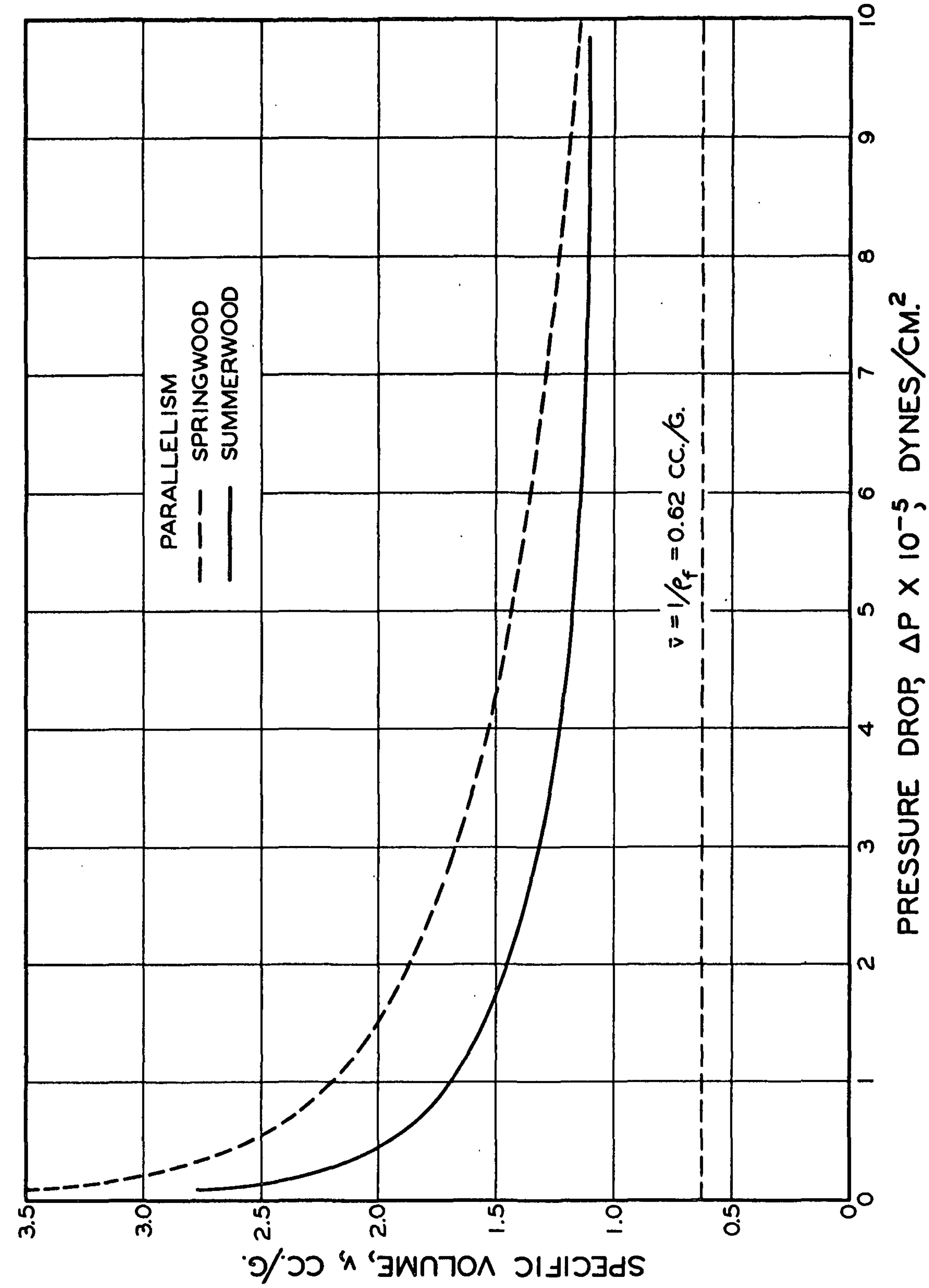

Figure 13. Parallelism of Specific Volumes for a Hypothetical Set of Fiber Samples

It had been anticipated that the relationship for the specific volumes of the springwood-summerwood fibers would give convergence over the entire schedule of pressure drop. Convergence represents the possible ease of collapse of the springwood fibers at the points of contact, with the largest $\Delta\bar{v}$ occurring for this fiber. It may be that although complete collapse is not occurring, the greater interactions along the transverse axis would produce an even greater change in $\bar{v}$ than collapse of contact points. Ultimately, of course, all values must approach as a limit the volume of pure cellulose, 0.62 cc./g. Convergence may also be explained by other combinations of mechanisms; the mechanisms cited here must be considered only as possibilities, and not necessarily the only mechanism involved.

Divergence is obviously a very complex interaction of different mechanisms for each of these two types of fibers. It would seem reasonable that with a thicker cell wall the summerwood fibers would resist the compressive deformation to a greater degree. However, if a greater stress intensification should result from a smaller contact area it may be possible that the unit stress would be great enough to cause fiber collapse. The springwood fibers may deform more readily under the fluid loading, and it may be possible that a fortuitous balance between increased area of contact and stress would yield a nearly constant unit stress for the case of the springwood.

Parallelism is difficult to rationalize on the basis of possible physical mechanisms. Possibly, for the case of fibers produced by such a mild action as the chloriting process, large enough pressure drops have not been encountered which would cause fiber collapse. This argument would lead to the conclusion that it would be necessary to investigate a much broader range of filtration conditions to yield higher point stresses. It is probable that the parallelism encountered in this investigation is the result of deficiencies in the theory and attempting to apply the concept of a pressure-dependent specific volume to a characterization wherein not high enough sensitivity is attainable to evaluate the differences, if any.

It appears then, that a variable specific volume defined in the hydrodynamic sense is not suitable to characterize some fiber property which may be related to conformability. As technology advances it may be possible to obtain the unstressed specific volume and a more rigorous solution for a variable term for $\bar{v}$ in the flow expression. Until these are at hand, however, the lack of sensitivity and required precision in the filtration test indicates that other avenues for characterizing fiber flexibility should be explored.

ACKNOWLEDGMENT

The contribution of the late Dr. Ingmanson, who participated in the original planning and experimental phase of the program, is hereby acknowledged.

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THE INSTITUTE OF PAPER CHEMISTRY

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APPENDIX I

CARLETRAN PROGRAM FOR VARIABLE SPECIFIC VOLUME

```
C       SPECIFIC VOLUME AND SURFACE FROM CONSTANT RATE EXPERIMENT DIMENSION
        T(9), DP(9), X(9), Y(9)
12      READ 1, F, E, R, V, CO, U
        DO 2 1=1, 9
2       READ 1, T(1)
        DO 3 1=1, 9
3       READ 1, DP(1)
        PRINT 1
        PRINT 1
        DO 4 1=1, 9
        C = F*DP(1)**E
        C1 = C**.5
        X(1) = C*C*C
        Y(1) = DP(1)/(C1*T(1))
        PRINT 1, T(1), DP(1), X(1), Y(1)
4       CONTINUE
        PRINT 1
        A = 3.5*(1.-E/2.)*V*R*CO*U*U
        B = 57.*(1.-E/2.)**6.
        Y1=Y(1)
        X1=X(1)
11      X2=X1+.00005
        SY=0
        DO 5 1=1, 9
        PX = 1000000.
        PXN = 1000000.
        DO 6 J=1, 9
        DX=X2-X(J)
        IF(I-J) 7,8,7
8       DX=1.
7       PX=PX*DX
        DXN=X(I)-X(J)
        IF(DXN) 9,10,9
10      DXN=1.
9       PXN=PXN*DXN
6       CONTINUE
        TI=(PX/PXN)*Y(1)
        SY=SY+TI
5       CONTINUE
        Y2=SY
        SV=((1.-Y1/Y2)/(B*(X2*Y1/Y2-X1)))**.33333
        SSV=((X2*Y1-X1*Y2)/(X2-X1)/A/SV**1.5)**.5
        SSW=SV*SSV
        P=(((X1+.000025)**.33333)/F)**(1./E)
        PRINT 1,X1,Y1,X2,Y2
        PRINT 1,P,SV,SSV,SSW
        X1=X2
        Y1=Y2
        IF(X1-X(9)) 11,11,12
        END
```

NOMENCLATURE

$\underline{T}$ = Time, seconds

$\underline{DP}$ = Pressure drop, dynes/cm.^{2}

$\underline{F}$ = Compressibility constant $\underline{M}$, c.g.s. units (g./cc.) (cm.^{2}/dyne)$^{\underline{N}}$

$\underline{E}$ = Compressibility constant $\underline{N}$, dimensionless

$\underline{R}$ = Filtrate density ρ, g./cc.

$\underline{V}$ = Filtrate viscosity ft., c.g.s. units

$\underline{CO}$ = Slurry consistency, g./cc.

$\underline{U}$ = Velocity, cm./sec.

$\underline{SV}$ = Specific volume, cc./g.

$\underline{SSV}$ = Specific surface, cm.^{2}/cc.

$\underline{SSW}$ = Specific surface, cm.^{2}/g.

APPENDIX II

AVERAGE SPECIFIC SURFACE AND SPECIFIC VOLUME

Specific surface and specific volume calculated by the integrated form of the Kozeny-Carman equation assuming linear fit of the filtration data:

	Specific Surface, SW, cm.^{2}/g.		Specific Volume, $\bar{v}$, cc./g.	
	Chlorited	Kraft	Chlorited	Kraft
Springwood	15,900	16,900	4.12	3.85
Summerwood	4,900	5,700	2.34	2.54

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